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TUNGSTEN — URANIUM DIOXIDE
CERMET CYLINDERS AT 3000° C

by Thomas J. Moore and Donald W. Adams

Lewis Research Center

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#### **ABSTRACT**

A procedure was developed to successfully join the longitudinal seam of a tungsten - uranium dioxide (W-UO $_2$ ) cermet cylinder by furnace brazing. Brazing was done in helium at  $3000^{\rm O}$  C with a tungsten - 25-weight-percent osmium (W-25Os) braze alloy. A moderate loss of tensile strength of the cermet parent material at 2500 $^{\rm O}$  C occurred due to the brazing cycle. The loss in strength was attributed to increased grain size of the tungsten matrix and possibly to migration of UO $_2$  along the grain boundaries of the tungsten matrix.

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#### SUMMARY

The objective of this program was to demonstrate the feasibility of applying a furnace brazing technique to join a tungsten - uranium dioxide (W-UO<sub>2</sub>) cermet for use at service temperatures to 2500°C The cermet used in this study contained 20 volume percent UO<sub>2</sub> and was in the form of a 0.020-inch(0.51-mm) wall cylinder, 1.12 inches (28.4 mm) in outside diameter by 1.5 inches (38.1 mm) long. A longitudinal seam butt joint was to be brazed. Complete joint fill with no defects and little or no excess braze was required. A tungsten - 25-weight-percent-osmium (W-25Os) braze alloy was the principal composition that was studied. Two other alloys were studied briefly: molybdenum - 5-weight-percent osmium (Mo-5Os) and tungsten - 50-weight-percent molybdenum - 3-weight-percent rhenium (W-50Mo-3Re).

Various furnace brazing techniques including methods of alloy placement and orientation of the axis of the W-UO $_2$  cylinder were studied. It was found that the cermet could be brazed successfully in helium at  $3000^{\circ}$  C using the W-25Os alloy.

Even though the  $3000^{\circ}$  C brazing temperature was above the melting point of UO $_2$  (about  $2800^{\circ}$  C) no significant reaction between the UO $_2$  and the tungsten matrix was observed. About a 20 percent loss of parent material strength at  $2500^{\circ}$  C occurred as a result of exposure to  $3000^{\circ}$  C for 1 minute. This loss of strength was attributed to an increase in grain size of the tungsten matrix and possibly to migration of UO $_2$  along the tungsten grain boundaries. A postbrazing heat treatment at  $2865^{\circ}$  C for 15 minutes was used successfully to eliminate the sigma phase in the W-25Os braze alloy. The loss of parent material strength at  $2500^{\circ}$  C due to the combined brazing plus postbrazing heat treatment was about 25 percent. Even though there was moderate loss in parent material strength, the feasibility of joining the longitudinal seam in a W-UO $_2$  cermet cylinder by furnace brazing was established.

## INTRODUCTION

The use of W-UO<sub>2</sub> cermets in fuel elements of high temperature nuclear reactors has been considered for possible space power and propulsion applications. In some applications, the cermet is a major structural material in the fuel element. The capability of joining such cermets affords increased freedom in fuel element design. Fusion welding of W-UO<sub>2</sub> cermets is not possible because the UO<sub>2</sub> particles vaporize far below the melting point of the tungsten. On the basis of an evaluation of six nonfusion welding methods, Moore and Watson (ref. 1) reported that gas tungsten-arc brazing, as developed by Japka and Gordon (ref. 2), was one of the more promising joining techniques for W-UO<sub>2</sub> for service in hydrogen at temperatures up to 2500° C. The most promising braze alloy was tungsten - 25-weight-percent osmium (W-250s). In tensile tests of butt joints brazed with W-250s, failure occurred in the parent material (ref. 1). Other alloys that showed promise were a tungsten - 50-weight-percent molybdenum - 3-weight-percent rhenium (W-50 Mo-3Re) and molybdenum - 5-percent osmium (Mo-50s) alloy. A drawback to the gas tungsten-arc brazing procedure, however, was the presence of excess braze metal at the joint.

The program presented in this report was limited in scope, consisting of only 10 brazing runs. Use was made of the braze alloys that were developed by Japka and Gordon, but furnace heating was used in place of the gas tungsten-arc process. By using furnace brazing it was anticipated that complete fill of the joint would be achieved with little or no excess braze metal at the joint. Thus, the main objective of this limited study was to determine whether furnace brazing is feasible in producing sound joints in W-UO<sub>2</sub> cermets. If this objective was met, brazing could be the first choice for joining the extremely difficult-to-weld cermet.

The joint brazed in this study was a longitudinal butt joint in a 1.12-inch (28.4-mm) diameter cylinder, 1.5 inches (38.1 mm) long, formed from sheet material 0.020 inch (0.51 mm) thick (see fig. 1). The tungsten cer met contained 20 volume percent (vol. %)  $\rm UO_2$  particles.

A number of furnace brazing variables were studied in a cursory manner. These variables were (1) composition, quantity and placement of the braze alloy, (2) positioning of cylinder during brazing, and (3) brazing time-temperature cycle variables. Primary emphasis was placed on a W-25Os brazing alloy, but the W-50Mo-3Re alloy and the Mo-5Os alloy were studied briefly. Brazing temperatures were about  $3000^{\circ}$  C for the W-25Os and W-50Mo-3Re alloys and  $2700^{\circ}$  C for the Mo-5Os alloy. A cermet brazed with the W-25Os alloy was subjected to postbrazing heat treatment at  $2865^{\circ}$  C in an attempt to eliminate the brittle  $\sigma$ -phase present in this braze alloy.

The results were evaluated primarily by visual and metallographic techniques. However, several  $2500^{\circ}$  C tensile tests were made on nonbrazed W-20 volume percent

 $\rm UO_2$  cermet sheet to determine the effects of the  $3000^{\rm O}$  C furnace brazing cycle and  $2865^{\rm O}$  C postbrazing heat treatment on the parent material. These brazing and heat treating temperatures were above the melting point of  $\rm UO_2$  ( $\sim\!2800^{\rm O}$  C, ref. 3).

#### MATERIALS

# Tungsten - 20 Volume Percent UO2 Cermets

Cylinders. - Manufacturing procedures for these cermets have been developed at the Lewis Research Center and are described by Watson in reference 4. Briefly, this process involved sintering and hot-rolling of W and UO<sub>2</sub> powder mixtures to produce fully dense cermet sheets. The faces of the cermet sheet used in the cylinders were clad with roll-bonded unfueled tungsten 0.0015 inch (0.040 mm) thick. Overall thickness of the clad cermet sheet was 0.020 inch (0.51 mm). The edges were not clad. As a first step in making the 1.12-inch (28.4-mm) outside diameter by 1.5-inch (38.1-mm) long cylinders, the end edges of the W-UO<sub>2</sub> sheets were ground square. Then the sheet was hot formed into a cylindrical shape as shown in figure 1.

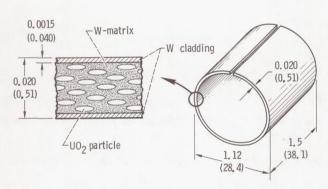
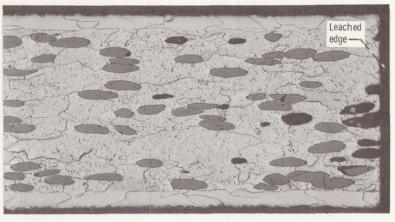


Figure 1. - W-UO $_2$  cylinder formed from sheet material. Dimensions are in inches (mm).

In preparation for brazing of the longitudinal seam, the cylinders were immersed in a warm  $(50^{\circ} \text{ C})$  solution of 50 percent nitric acid (by volume) for 15 minutes. This treatment removed the  $\text{UO}_2$  particles which were exposed at the edges of the sheets (fig. 2). In this way problems associated with a mixture of  $\text{UO}_2$  and molten braze metal during the brazing cycle were minimized.

<u>Tensile specimens</u>. - Cermet sheet material (0.020 inch (0.51 mm) thick) was used for preparation of the tensile specimens. It was necessary to obtain specimens from several different lots of cermet sheet because this material was in short supply. The procedure for fabricating the sheets for tensile specimens was the same as that used for



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Figure 2. - W-UO<sub>2</sub> cermet sheet with leached edge. Longitudinal section; Murakami's reagent. X100.

preparing sheets for the W-UO<sub>2</sub> cylinders. Although none of the cermet sheets for tensile specimens were made from the same lot of starting materials used to fabricate cylinders, the basic composition of the cermets was the same, W-20 volume percent UO<sub>2</sub>.

# Braze Alloys

Selection of braze alloys. - Braze alloy selection was based on the results of an evaluation study by Moore and Watson (ref. 1) in which it was shown that tensile failures occurred in the parent material for joints brazed with W-25Os. Other alloys reported to show promise (ref. 1) were W-50Mo-3Re and Mo-5Os. Nominal compositions, melting point, and brazing temperatures for these braze alloys are shown in the following table.

Braze alloy, wt. %	Temperature at initiation of melting (ref. 2), OC	Brazing temperature, <sup>o</sup> C	
W-250s	2975	2985 to 3040	
W-50Mo-3Re	2855	3025	
Mo-5Os	2595	2700	

Metal powders. - Metal powders of the elements in the braze alloys were procured from commercial vendors. The chemical analysis provided by the supplier and the average particle size of raw materials used in preparing the braze alloys are as follows:

Material	Average particle size, $\mu m$	Nominal purity, percent	
W	$-4\frac{1}{2}$	99.9	
Mo	$-4\frac{1}{2}$	99.9	
Os	74 99.85		
Re	44 to 74	99.9999	

In an attempt to minimize adsorbed oxygen, the powders were heated in a container to  $650^{\circ}$  C in flowing hydrogen. The outlet gas was analyzed until the  $\rm H_2O$  content of the hydrogen was less than 50 ppm. The container was then sealed and transferred to a dry box. The dry box was evacuated to  $1\times10^{-4}$  torr  $(1.33\times10^{-2}~\rm N/m^2)$  and backfilled with high purity argon containing <4 ppm  $\rm O_2$ , <7 ppm  $\rm N_2$ , <1.5 ppm  $\rm H_2O$ , and <1 ppm hydrocarbons. The container was then opened and the powders were weighed for preparation of the required powder mixtures. The mixtures were then removed from the dry box and compacted into 10 gram pellets using a 1/2-inch (1.27-cm) diameter die and a hydraulic press. The powder mixtures prepared for each braze alloy were listed below along with the actual alloy composition after melting.

Nominal alloy composition	Actual weight percent in prepared mix	Final braze alloy analysis, wt. %	
W-25Os W-50Mo-3Re	W-27Os W-50Mo-3Re (same as aim)	W-24.5Os 46.4W-49.86Mo-3.58Re	
Mo-5Os	Mo-6Os	Mo-5.1Os	

The Os contents of the W-25Os and Mo-5Os alloys in the powder mixtures are purposely made slightly higher than the nominal composition to compensate for Os vaporization losses during melting.

The alloy pellets were transferred to a welding chamber which was evacuated to  $1\times10^{-5}$  torr  $(1.33\times10^{-3} \text{ N/m}^2)$  and backfilled with argon.

Melting. - A conventional gas tungsten-arc welding torch was used to melt the alloy pellets. The argon atmosphere in the welding chamber was provided from a liquid argon supply. It contained the following impurities in ppm:  $O_2$ , <10;  $N_2$ , <5; H, <40; total impurities, <50. The dew point of the argon was -62°C at 1 atmosphere. Just prior to melting, the argon was gettered by striking an arc on zirconium. During melting of the braze alloys, the pellets rested in a dished cavity in a large copper chill block. The

pellets were triple melted. One side of the pellet was melted to a button-like form; then, the other side was melted; and finally, the first side of the button was melted again. The first sample buttons of each braze lot were cut in two and metallographically examined. All buttons examined were completely melted and homogeneous.

<u>Crushing</u>. - The arc-melted buttons were crushed first by a hand pestle and mortar to approximately 0.07-inch (1.7-mm) particles. The particles were then ground in a steel ball mill to produce a powder having an average particle size of about 0.003 inch (74 micron) (-200 mesh). The braze alloy particles were treated with HCl in order to remove all traces of iron. Following the acid treatment the powders were washed in water and then in isopropyl alcohol.

### APPARATUS AND PROCEDURE

# Brazing Furnace

Brazing was done using the induction-heated cold-wall furnace shown in figure 3. The working zone inside the tungsten susceptor is 2.1 inches (53 mm) in diameter by 3.0 inches (76 mm) in length (fig. 3, inset). For each run the samples were placed in the brazing furnace; after a vacuum of about  $1\times10^{-4}$  torr (1.33×10<sup>-2</sup> N/m²) was attained, the furnace was backfilled with helium until the pressure in the chamber was about 10 millimeters of mercury (1.33×10<sup>3</sup> N/m²) below atmospheric. Thus, with helium pressure in the furnace of just under 1 atmosphere, losses of UO<sub>2</sub>, Os, Mo, and Re by way of vaporization were minimized. The gas pressure in the furnace was not allowed to go any higher for safety reasons. In order to reduce thermal stresses on cooling from the brazing temperature, a cooling arrest for 1 hour at 900° C was used for all runs. Impurity content of the 99.995 percent helium, grade A, was as follows in ppm: O<sub>2</sub>, <2; Ar, Ne, H, CO<sub>2</sub>, and CO, <37; moisture, <10 (dew point, -61° C).

# Temperature Measurement

Specimen temperature during the brazing cycle was measured with an optical pyrometer (fig. 3). Since sighting was made through a prism and a window in the top of the furnace, a correction was made using routine pyrometer techniques.

In the run where brazing was done with the cylinder axis in a vertical position, the temperature was read by sighting into a blackbody hole in a tungsten bar located next to the cylinder.

For most of the runs where the cylinder axis was horizontal, an attempt was made



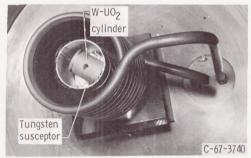


Figure 3. – Cold-wall induction-heated brazing furnace with close-up of W-UO $_2$  cylinder in position for brazing.

to detect flow of the braze metal on the inside diameter surface by sighting directly onto the braze joint through a 0.19-inch (4.8-mm) diameter hole in the side of the cylinder. Since the glare was intense inside the cylinder, flow of the braze metal could not be detected. The brightness of this glare was read as the temperature.

It is estimated that the corrected optical pyrometer readings are within about ±1 percent of the true temperature.

# Fit-Up and Joint Alinement

A prebrazing stress relief heat treatment was used in order to improve fit-up and joint alinement. For this heat treatment, the W-UO $_2$  cylinders were tightly wrapped with 0.010-inch (0.25-mm) diameter tungsten wire, heated to  $1650^{\circ}$  C at  $1\times10^{-5}$  torr (1.33×10<sup>-3</sup> N/m $^2$ ), and held at temperature for 15 minutes. After this heat treatment,

the residual gap opening with the wires removed was less than 0.010 inch (0.25 mm).

In order to maintain a tight gap during brazing of the longitudinal seam butt joint, two 0.010-inch (0.25-mm) diameter tungsten wires were tightly wrapped around the cylinder, one near each end. These wires maintained a tight gap for capillary flow of the braze metal.

# Brazing Procedure

The furnace brazing program consisted of 10 runs. A number of changes were made in the procedure from run to run. The variations in procedure are shown below for eight brazing runs with the W-25Os alloy. None of the variables were examined thoroughly because of the limited nature of the program.

- (1) Quantity and placement of the braze alloy
  - (a) Method of placement of the braze alloy, as a dry powder or in a lucite (methyl methacrylate) slurry
  - (b) Quantity of braze alloy, 0.015 to 0.25 gram
  - (c) Placement of braze alloy, inside diameter or outside diameter surface
- (2) Positioning of cylinder in the furnace
  - (a) Method of support, standing on plates or wire suspension
  - (b) Orientation of cylinder axis, horizontal or vertical
  - (c) Orientation of joint for horizontal cylinder, 6 or 12 o'clock
- (3) Brazing time-temperature cycle variables
  - (a) Heating time to braze temperature, 10 to 23 minutes
  - (b) Brazing temperature, 2985° to 3040° C
  - (c) Time at brazing temperature, 0.5 to 15 minutes
  - (d) Postbrazing heat treatment, 2865° C for 15 minutes (for one run)

One braze joint was made with the Mo-5Os alloy and one joint with the W-50Mo-3Re alloy. The brazing conditions are as follows:

Condition	Mo-5Os	W-50Mo-3Re	
Quantity and placement of braze alloy:			
Method of placement	Slurry	Dry powder	
Quantity of braze alloy			
Placement of braze alloy	Outside-diameter surface	Inside-diameter surface	
Positioning of cylinder in the furnace:			
Method of support	Wire suspension	Wire suspension	
Orientation of cylinder axis	Horizontal	Horizontal	
Orientation of joint	6 o'clock	6 o'clock	
Brazing time-temperature cycle variables:			
Heating time to braze temperature, min	20	12	
Braze temperature, °C	2700	3025	
Time at braze temperature, min	1	1	
Postbrazing heat treatment	None	None	

# Evaluation

<u>Braze joints</u>. - The brazed joints were evaluated primarily by visual and metallographic techniques. Neither tensile tests or simulated service heat treatments were applied to the furnace brazed joints.

Tensile tests. - In 2500°C tensile tests, Moore and Watson (ref. 1) showed that failure occurred in the W-UO<sub>2</sub> parent material for butt joints brazed with the most promising alloy, W-25Os. Therefore, tensile tests in this study were limited to determining the effects of the brazing temperature cycle on parent material strength. Two different heat treatments were given to unbrazed cermet sheets (0.020 in. (0.51 mm) by 3/4 in. (19.0 mm) by 6 in. (152 mm)) in flowing helium: (1) 3000°C for 1 minute to simulate a typical brazing cycle; or (2) 3000°C for 1 minute plus 2865°C for 15 minutes to simulate a brazing and heat treating cycle used with the W-25Os braze alloy.

Triplicate tensile specimens were machined from 3/4-inch (19.0-mm) by 6-inch (152-mm) blanks in the as-received condition and duplicate tensile specimens were prepared in each of the above heat treated conditions. Electrical-discharge machining procedures were used to obtain a 3/8-inch (9.5-mm) wide by  $1\frac{1}{4}$ -inch (31.8-mm) long reduced section. The tensile specimens were tested at  $2500^{\circ}$  C in vacuum,  $3\times10^{-5}$  torr  $(4\times10^{-3} \text{ N/m}^2)$ , at a strain rate of 0.03 inch per minute (0.76 mm/min) using equipment and procedures described in reference 5.

#### RESULTS AND DISCUSSION

#### Brazed Joints

W-25Os braze alloy joints. - Brazing studies were concentrated on the W-25Os alloy. The best results from eight brazing runs with the W-25Os alloy were obtained using the support and braze alloy placement method shown in figure 4. To prevent the de-

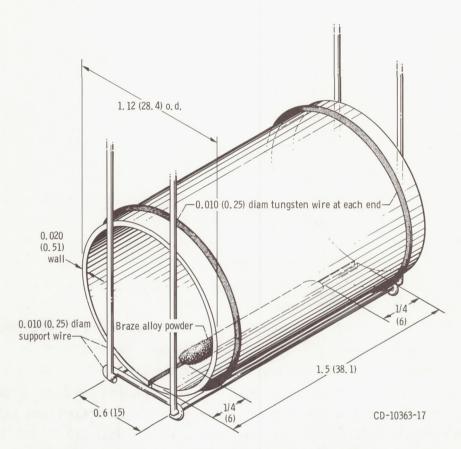
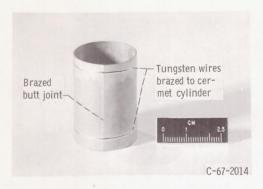
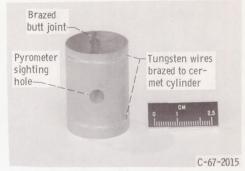


Figure 4. - Method of specimen support and braze alloy placement. No braze alloy is placed within 1/4 inch (6 mm) of ends in order to prevent erosion at ends of joint. Dimensions are in inches (mm).

velopment of small slots at the ends of the joint due to erosion, no braze metal powder was placed close to the ends. Photographs showing the outside diameter and a portion of the inside diameter of a successfully brazed W-UO $_2$  cylinder are shown in figure 5. The external appearance of this joint was excellent in that complete joint fill was achieved with no excess braze metal at the joint. As was the case with all of the brazed cylinders, however, the tungsten wires were brazed to the W-UO $_2$  cylinder. The brazing conditions





(a) Outside-diameter surface of brazed joint.

(b) Portion of inside-diameter brazed joint and sighting hole.

Figure 5. -  $W-UO_2$  cermet cylinder brazed with W-25 Os alloy. Complete fill of longitudinal butt joint was achieved with little or no excess braze metal at joint.

that gave the best results are shown below. Even though this procedure gave satisfactory results more work may be necessary to fully evaluate the effect of the variables listed.

Furnace brazing procedure developed for the W-25Os braze alloy. - The furnace brazing procedure is as follows:

Alloy composition, W-25Os

Method of placement of the braze alloy, dry powder

Quantity of braze alloy, 0.25 gram

Placement of braze alloy, inside diameter surface (see fig. 4)

Method of support, wire cradle (see fig. 4)

Orientation of cylinder axis, horizontal

Orientation of joint, 6 o'clock

Heating time to braze temperature, 20 min

Brazing temperature, 3010° C

Brazing atmosphere, helium (10 mm of Hg (1.33×10<sup>3</sup> N/m<sup>2</sup>) below atmospheric)

Time at brazing temperature, 1 min

Postbrazing heat treatment, 2865° C for 15 minutes (as an integral part of the brazing cycle)

A photomicrograph from a joint that was brazed and heat treated using the above procedure is shown in figure 6. This joint exhibits a single, alpha ( $\alpha$ )-phase microstructure. Both the brazing and heat treat temperatures were above the melting point of  $\rm UO_2$  (about 2800°C). Coalescence took place between the braze metal and the tungsten matrix of the W-UO<sub>2</sub> composite sheet and there was no braze metal cracking. Joint aline ment is excellent. With the results of this run, the principal objective of the program was achieved; that is,  $3000^{\circ}$  C furnace brazing of a longitudinal butt joint in a W-UO<sub>2</sub> cermet cylinder is feasible.

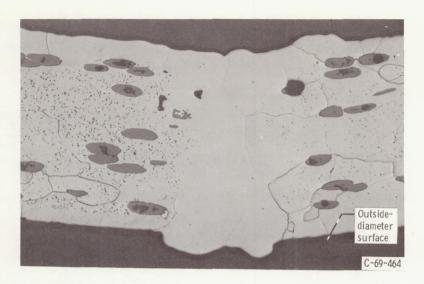


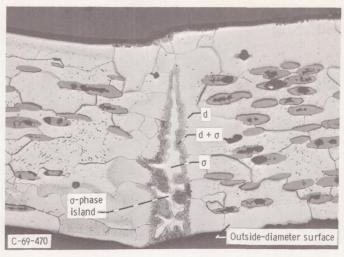
Figure 6. - Joint in W-UO<sub>2</sub> cermet cylinder brazed with W-25 Os braze metal. This brazed joint exhibits single-phase microstructure after being exposed to postbrazing heat treatment at 2865° C for 15 minutes after 1-minute hold time at 3010° C for brazing. Murakami's reagent. X100.

Without the postbrazing heat treatment, the W-25Os braze metal has a two-phase microstructure, alpha ( $\alpha$ ) + sig ma ( $\sigma$ ). A cross section of such an as-brazed joint is shown in figure 7(a). This microstructure, which contains  $\sigma$ -phase, is undesirable because  $\sigma$ -phase is crack-sensitive as shown in figure 7(b). Here, a microcrack exists in a  $\sigma$ -phase island. The results of a hardness traverse (shown below) reveals the  $\sigma$ -phase is extremely hard in comparison with  $\alpha$ -phase. The tungsten matrix of the W-UO<sub>2</sub> base material is much softer than either phase.

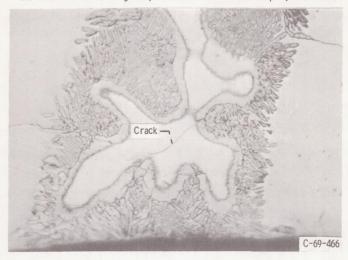
DPH hardness, kg/mm<sup>2</sup>

Tungsten matrix of	W-25Os braze metal		
W-UO <sub>2</sub> base material	α-phase	σ-phase	
340	530 to 820	over 1370	

The vee-shaped region of the braze alloy in figure 7(a) illustrates the fact that the braze metal fills a vee joint. If the joint edges had been square, much less volume of braze metal would have been in the joint. With less braze metal, the amount of crack-sensitive  $\sigma$ -phase in the W-25Os braze metal would be reduced by diffusion between the smaller amount of braze metal and the tungsten matrix of the cermet. Some misaline-



(a) Cross section showing two-phase braze metal in vee-shaped joint. X100.



(b) Crack in  $\sigma$ -phase island. X500.

Figure 7. - As-brazed joint in W-UO $_2$  cermet cylinder brazed with W-25 Os braze alloy.

ment is evident at the joint in figure 7(a), but the braze alloy has feathered-in the protruding edges of the cermet.

The hold time at the brazing temperature was the variable for another W-25Os braze joint. In this case the specimen was held at  $3040^{\circ}$  C for 15 minutes instead of the usual 1 minute. This was done in order to transform the  $\sigma$ -phase to  $\alpha$ -phase. The UO<sub>2</sub> particles, which melt at about  $2800^{\circ}$  C, were probably molten during brazing. This brazing procedure was successful in that the  $\sigma$ -phase was eliminated, but an undesir-

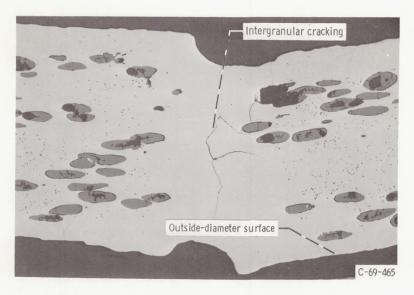


Figure 8. - Joint in W-UO<sub>2</sub> cermet cylinder brazed with W-25 Os braze metal.

Intergranular cracks appear in single-phase W-25 Os braze metal as result of reactions that took place during 15-minute hold time at 3040° C brazing temperature.

Unetched. X100.

able side effect was produced in which the development of intergranular cracks occurred, as shown in the unetched condition in figure 8. This is the only time that intergranular cracking was seen in the W-25Os braze alloy. This cracking may be essentially a shrinkage void. The cracking might also have resulted from intergranular penetration of  $\rm UO_2$ . A combination of metallurgical and mechanical factors are probably involved. An interesting observation from this brazing run was that the entire outside-diameter and inside-diameter surfaces were wet by the braze metal. It appears that the lower postbrazing heat treatment (2865 $^{\rm O}$  C for 15 min) is preferable for eliminating the  $\sigma$ -phase.

One brazing run was made with the cylinder axis in a vertical position. In this case, a slurry of the W-25Os braze alloy was placed at the outside-diameter surface along the joint. The brazing operation produced unsatisfactory results because the braze metal eroded the sidewalls of the joint and produced an open gap in the lower half of the joint.

The structural change associated with brazing at  $3000^{\circ}$  C (above the melting point of  $UO_2$ ) can be noted by comparing the as-fabricated structure (fig. 2) to that after brazing (figs. 6, 7(a), and 8). The voids within the  $UO_2$  particles shown in figures 6, 7(a), and 8 probably represent shrinkage voids since  $UO_2$  is molten above  $2800^{\circ}$  C. In the molten state the  $UO_2$  particles would tend to expand and increase the size of the cavity that contains them. On cooling, there would be insufficient material to fill the larger cavity thus producing shrinkage voids. Part of the void area may be attributed to metallographic pullout.

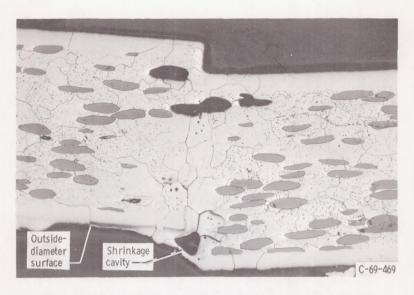


Figure 9. - Joint in W-UO<sub>2</sub> cermet cylinder brazed with Mo-5 Os braze metal. A shrinkage cavity is apparent in single-phase Mo-5 Os braze alloy. Extensive grain growth across bond lines attests to complete metallic bonding between braze metal and cermet. Murakami's reagent. X100.

Mo-5Os braze alloy joint. - One joint was brazed with the Mo-5Os braze alloy. Complete fill of the joint by the braze alloy was not obtained for most of the joint length. A cross section where joint fill was adequate is shown in figure 9. Coalescence with the cermet has occurred. But a shrinkage cavity is present and alinement is poor. (The oval voids in the cermet are areas where UO<sub>2</sub> particles are believed to have been lost during preparation for metallographic examination.)

Since the brazing with the Mo-5Os alloy was achieved below the melting point of  $UO_2$  (2700°C), no structural change took place in the cermet compared to previous brazing runs made at  $3000^{\circ}$  C with W-25Os. This effect can be seen by comparing figure 9 with figures 6, 7(a), and 8.

W-50Mo-3Re braze alloy joint. - A single joint was also made using the W-50Mo-3Re braze alloy. A photomicrograph of this joint is shown in figure 10. The original interfaces between cermet and braze metal have been obscured by grain growth across the bond lines. However, there was some braze metal porosity at other areas of the joint (not shown). The properties of the W-50Mo-3Re alloy as a braze metal were good in that complete fill of the joint was obtained and there was coalescence between braze metal and cermet.

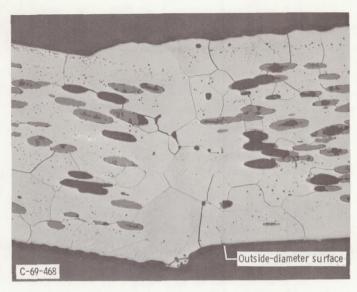


Figure 10. - Joint in W-UO<sub>2</sub> cermet cylinder brazed with W-50Mo-3Re braze metal.

Coalescence between single-phase W-50Mo-3Re braze metal and cermet is complete. Murakami's reagent. X100.

### Tensile Tests

The results of 2500°C tensile tests on the cermet sheet are shown in table I. Note that there is about 20 percent loss of tensile strength as a result of the simulated 3000°C braze cycle when compared to strength of as-fabricated cermet sheet. A 3000°C simulated braze cycle plus a 15-minute exposure to 2865°C produced a loss in strength of about 25 percent in comparison with as-fabricated cermet sheet.

Two factors are probably involved in the weakening that was observed in the heat-treated cermet sheet. Grain growth in the tungsten matrix during exposure to temperature near  $3000^{\circ}$  C is believed to be one factor. It has been clearly shown (refs. 5 and 6) that large-grained-tungsten sheet material is considerably weaker than finer grained sheet at temperatures between  $2100^{\circ}$  to  $2750^{\circ}$  C. In addition, migration of molten  $UO_2$  along the grain boundaries of the tungsten matrix can also weaken the cermet. A major weakening effect due to migration of  $UO_2$  was not of major concern, however, because Eash (ref. 7) has shown relatively minor attack of tungsten by molten  $UO_2$  in samples that were held at  $3000^{\circ}$  C for 168 hours.

# TABLE I. - TENSILE STRENGTH OF 0.020 INCH (0.51 MM) TUNGSTEN - 20-VOLUME-PERCENT URANIUM DIOXIDE CERMET SHEET AT 2500<sup>0</sup> C BEFORE AND AFTER EXPOSURE TO SIMULATED BRAZING AND

#### BRAZING-PLUS-HEAT-TREATMENT CYCLES

[Environment, vacuum of  $3\times10^{-5}$  torr  $(4\times10^{-3} \text{ N/m}^2)$ .]

Condition	Specimen	Ultimate tensile strength		Average ultimate tensile strength	
		psi	N/mm <sup>2</sup>	psi	N/mm <sup>2</sup>
As fabricated	1	3070	21.2	3340	23.0
	2	3380	23.3		
	3	3580	24.7		
Heat treated at 3000° C	4	2480	17.1	2710	18.7
for 1 min in helium	5	2940	20.3		
Heat treated at 3000° C	6	2360	16.3	2490	17.2
for 1 min in helium and $2865^{\circ}$ C for 15 min in helium	7	2620	18.1		

# CONCLUSIONS

- 1. The longitudinal seam of a tungsten uranium dioxide  $(W-UO_2)$  cermet cylinder can be successfully butt-brazed in a furnace at  $3000^{\circ}$  C in helium using a tungsten 25-weight-percent-osmium (W-25Os) braze alloy.
- 2. The best brazing results were obtained with braze alloy particles placed along the joint on the inside diameter of the W-UO $_2$  cermet cylinder. The axis of the cylinder was in a horizontal plane and the joint was in the 6 o'clock position.
- 3. The crack-sensitive  $\sigma$ -phase in the W-25Os alloy can be converted to  $\alpha$ -phase by including a postbraze diffusion heat treatment at 2865 $^{\circ}$  C for 15 minutes.
- 4. Brazing at  $3000^{\circ}$  C produced about a 20-percent loss in the strength of the W-UO<sub>2</sub> parent material at  $2500^{\circ}$  C. The loss in parent material strength was attributed to increased grain size of the tungsten matrix and possibly to migration of UO<sub>2</sub> along the tungsten grain boundaries.
- 5. Single runs made with molybdenum 5-weight-percent osmium (Mo-5Os) and tungsten 50-weight-percent molybdenum 3-weight-percent rhenium (W-50Mo-3Re) braze alloys have shown that both of these alloys also are promising for furnace brazing the W-UO<sub>2</sub> cermet.

#### RECOMMENDATIONS FOR FUTURE WORK

- 1. Although the W-25Os braze alloy was successfully used in this study, additional studies on lower melting point braze alloys would be desirable. A lower brazing temperature would reduce grain growth in the tungsten matrix of the W-UO $_2$  cermet. Brazing below about 2800 $^{\rm O}$  C would eliminate possible reaction between the molten UO $_2$  and the tungsten matrix. In a modified W-25Os braze alloy, a further objective would be to avoid compositions that would result in the formation of sigma phase.
- 2. Mating edges should be ground to form a square butt joint. This would avoid the V-type joint which was encountered on this program and thus require less braze metal in the joint. With less braze metal the amount of σ-phase with W-25Os braze metal would be reduced greatly by diffusion between the smaller amount of braze metal and the tungsten matrix of the cermet. Use of a close-fitting square butt joint might eliminate the need for a high-temperature post-brazing heat treatment.
- 3. Better mechanical means of maintaining the gap for brazing should be devised. The wire-wrapping technique used on this program would probably require that the cylinders be made over-length. Then after brazing the wire-wrapped ends would be cut off in a final sizing operation.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, February 3, 1969, 122-28-02-04-22.

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